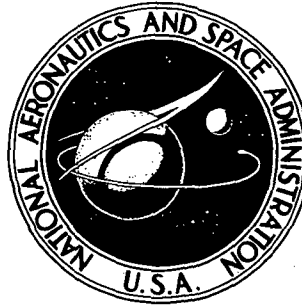


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EFFECT OF TEMPERATURE ON TENSILE
AND CREEP CHARACTERISTICS
OF PRD49 FIBER/EPOXY COMPOSITES

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16. Abstract <p>Tensile and creep data of PRD49-I and -III fiber/epoxy-resin composites are presented. Tensile data were obtained from 20 to 477 K (-423° to 400° F). Tensile strengths and moduli were determined at selected temperatures. Creep data are presented for fiber composites at 297, 422, and 450 K (75°, 300°, and 350° F) for as long as 1000 hours at stress levels of approximately 50 and 80 percent of the ultimate tensile strength at 297 K (75° F). Details of tensile specimens and test procedures used in the investigation are presented.</p>					
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EFFECT OF TEMPERATURE ON TENSILE AND CREEP CHARACTERISTICS OF PRD49 FIBER/EPOXY COMPOSITES

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SUMMARY

An experimental investigation was conducted to determine the strength and creep characteristics of PRD49 fiber in an epoxy matrix. Properties were determined at selected temperatures in the range from 20 to 477 K (-423° to 400° F).

Tensile strength properties were generally retained to 450 K (350° F); however, at 477 K (400° F) the tensile strength was about 73 percent of that at 297 K (75° F). The tensile modulus showed no significant change at elevated temperatures; however, at 20 K (-423° F) the modulus increased about 40 percent compared to the modulus at 297 K (75° F).

In creep testing, the PRD49 fiber experienced an accelerated primary creep followed by a much lower secondary creep rate. At room temperature, the fiber exhibited a low secondary creep rate and sustained a tensile stress of about 80 percent of the ultimate tensile strength for 1000 hours without failure. Humidity had only a minor effect on the creep behavior of the fiber.

INTRODUCTION

DuPont's recently developed organic fiber, PRD49, has been reported to have excellent reinforcing capabilities in structural composites (refs. 1 and 2). The fiber has high tensile strength and modulus and a low density that yields specific properties that compare favorably to those of currently available high-performance fibers such as graphite, boron, and glass. Results of short-term tensile tests have shown that the PRD49 fiber retains 75 percent of the room-temperature tensile strength at 477 K (400° F) and that both the tensile modulus and elongation are reduced 18 percent (ref. 1). After 450 hours exposure at 514 K (465° F) in air, the fiber retains 70 percent of the room-temperature tensile strength. The fiber has also shown low creep properties to

1000 hours duration at room temperature (ref. 1). Data on the creep characteristics of the fiber at elevated temperatures, however, are limited.

The present investigation was conducted to study the elevated-temperature creep characteristics of unidirectional PRD49/epoxy composites at various stress levels. Tensile strength and modulus of the composites were also determined at temperatures ranging from 20 to 477 K (-423° to 400° F).

MATERIALS AND FABRICATION

PRD49 fiber types I and III were used in this investigation. Typical properties of the fibers are listed in table I. The yarn area used in calculating the fiber stress was based on the weight per unit length divided by the fiber density. The resin matrix formulation (ref. 3) and cure cycle are also noted in table I. The formulation was chosen on the basis of its high-deformation temperature.

TABLE I. - TYPICAL PROPERTIES OF PRD49 FIBER

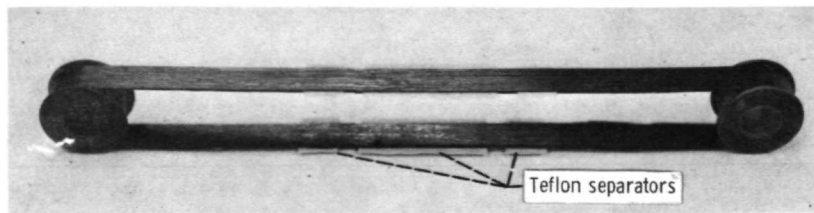
Fiber ^a	Specific gravity	Yarn area ^b		Tensile strength ^c		Tensile modulus ^c	
		cm ²	in. ²	N/cm ²	psi	N/cm ²	psi
PRD49-I	1.47	28.4×10 ⁻⁵	4.4×10 ⁻⁵	235×10 ³	340×10 ³	14.5×10 ⁶	21.0×10 ⁶
PRD49-III	1.45	32.3×10 ⁻⁵	5.0×10 ⁻⁵	277×10 ³	400×10 ³	13.1×10 ⁶	19.0×10 ⁶

^aE. I. DuPont de Nemours and Company. Yarn impregnated with Union Carbide Corp. ERLB 4617 and Furane Plastics Inc. hardener 9247 (22 phr). Cure cycle: 2 hr at 355 K (180° F) and 16 hr at 450 K (350° F).

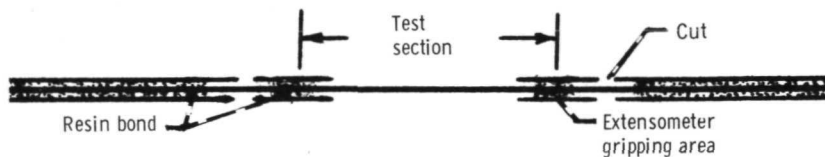
^bCalculated cross-sectional area of yarn.

^cTypical properties reported by manufacturer at 297 K (75° F).

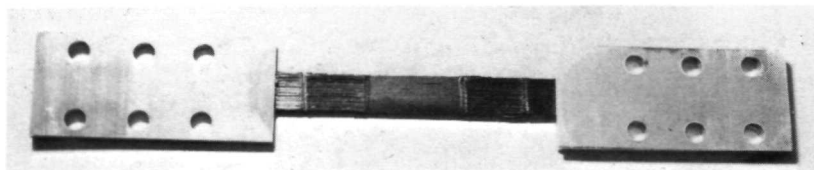
Before fabrication, the fiber was dried at 339 K (150° F) for 16 hours. Two types of specimens were used to investigate tensile and creep properties. Both specimens were made by using a filament-winding technique. Tests of the PRD49-I were made with a loop specimen in which 100 turns of yarn were wound over two end-spools in three layers and impregnated with the epoxy resin. The resulting specimen consisted of two parallel bands with a known fiber count bonded to end-spools in a manner similar to that shown in figure 1(a). The loop specimen was developed with the intent of minimizing the usual gripping problems encountered in testing a uniaxial fiber/resin composite tensile specimen. Although the loop concept proved adequate as a tensile and creep specimen at normal temperature, the specimen was found to be limited in performance at elevated temperatures.



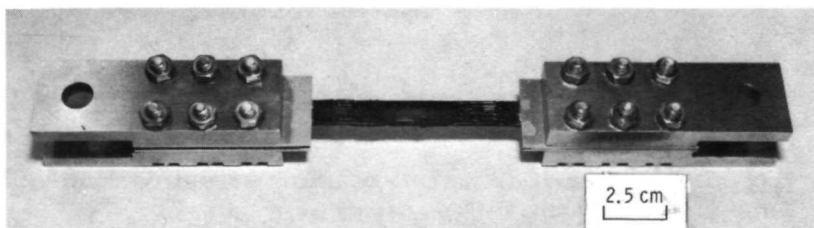
(a) Three-layer filament-wound specimen with teflon separators.



(b) Schematic of removed outer fiber reinforcement at test section.



(c) Aluminum-tabbed tensile specimen showing test section and extensometer mounting pads.



(d) Assembled specimen with tensile grips.

Figure 1. - Development of a filament-wound tensile specimen with in-situ end reinforcements.

Because of the limitation encountered with the loop specimen, a unidirectional tensile specimen was developed for continued testing of PRD49-III composites. The fabrication details of this specimen are shown in figure 1. The specimen was produced from a filament-wound preform as shown in figure 1(a). It was reasoned that the filament-wound preform would provide in-situ wound end reinforcement of the tensile specimen and also provide a reinforced area for the extensometer grips. Basically, the preform consisted of three 1.27-centimeter by 25.4-centimeter (1/2-in. by 10-in.) unidirectional layers filament-wound about the end spools. As shown in figure 1(a), the outer fiber layers were separated from the middle layer by means of thin Teflon-coated glass-cloth separators which were placed between the individual layers during the winding process.

The separators isolated the reinforced extensometer gripping area and the test section from the end reinforcement.

The center separator cloth, approximately 5.08 centimeters (2.0 in.) long, established the gage section of the final specimen. After the preform was cured, the composite was removed from the end spools and sectioned into two separate flat strips approximately 22.8 centimeters (9.0 in.) long. The outer layers were carefully removed from the gage section area by saw cuts exposing the middle layer. Additional cuts were also made to release the extensometer gripping area from the end reinforcement as shown in figure 1(b). Further reinforcement of the tensile specimen ends was provided by aluminum tabs that were adhesively bonded to the unidirectional composite (fig. 1(c)). The aluminum tabs also provided a means of attaching pinned grips (fig. 1(d)) that adapt to either tensile or creep testing machines.

In addition to providing the in-situ end reinforcements of the tensile specimen, the filament-winding fabrication process also provides essentially perfect collimation of the fibers. Another advantage of the filament-wound specimen is that no longitudinal cutting is required to establish the width of the specimen. Cutting unidirectional tensile specimens from flat laminates unavoidably results in some discontinuous fibers along the cut edges of the specimen.

APPARATUS AND PROCEDURE

Tensile Tests

Tensile tests were performed to establish tensile strength and modulus at various temperatures from 20 to 477 K (-423° to 400° F). Cryogenic tensile tests were conducted by immersion of the specimens in either liquid hydrogen for the 20 K (-423° F) tests or liquid nitrogen for the 77 K (-320° F) tests. Elevated-temperature tests were performed in an environmental oven that was equipped with a thermocouple probe located in the proximity of the test section for temperature control. The tests were performed in about 15 minutes after the temperature had stabilized. All tests were run at a cross-head loading rate of 1.27 millimeters (0.05 in.) per minute. Strain was measured at all temperatures by means of resistance strain gages that were adhesively bonded to the specimen test section. Tensile tests were performed on the continuous loop specimens of PRD49-I and on the unidirectional tensile specimen of PRD49-III described previously.

Creep Tests

Creep tests were performed in static loading machines equipped with lever arms that provided a 20:1 ratio of specimen load to deadweight load. The machines were provided with a heating chamber to maintain constant temperature throughout the duration of the test. The temperature was monitored by a thermocouple attached to the test section of the specimen. Creep tests were performed at 297, 422, and 450 K (75°, 300°, and 350° F). The stress levels were approximately 50 and 80 percent of the ultimate short-time tensile strength of the composite determined at the test temperatures.

Creep measurements were made on loop specimens of PRD49-I and on unidirectional tensile specimens of PRD49-III. Creep values were obtained from loop specimens by applying a correction factor to the overall elongation of the specimen. The correction factor was determined from the ratio of the unit strain, measured by a strain gage, to the corresponding overall elongation measured during a tensile test.

The creep of PRD49-III was measured by means of a clamp-on extensometer with the grips attached to the reinforced areas of the specimen, as described in the section MATERIALS AND FABRICATION. The grips were set at a 5.08-centimeter (2.0-in.) gage length. Deformation of the test section was transferred from the grips to the outside of the testing chamber by means of rod-and-tube extensions. A linear, variable, differential transformer (LVDT) was located outside the chamber and attached to the rod-and-tube extensions. The calibrated output from the LVDT was traced on a strip-chart recorder. The time was recorded by an accumulative counter that automatically shut off upon failure of the specimen.

DISCUSSION AND RESULTS

Tensile Properties

PRD49 fiber is a unique organic fiber in that the stress-strain relation is linear to its ultimate tensile strength. This behavior was observed for both type I and type III fibers throughout the temperature range from 20 to 477 K (-423° to 400° F). Figures 2 and 3 show the stress-strain relation at selected temperatures. In this investigation stress is presented in terms of fiber stress because of the precisely known fiber content wound into the loop and unidirectional tensile specimens. The fiber stress was determined by dividing the load by the total fiber area. The strength contributed by the resin can be considered negligible (about 1.0 percent) because of the high fiber-to-resin-modulus ratio. The average fiber tensile strength values at 297 K (75° F) were 221 000 N/cm² (320 000 psi) and 294 000 N/cm² (425 000 psi) for the type I and type III

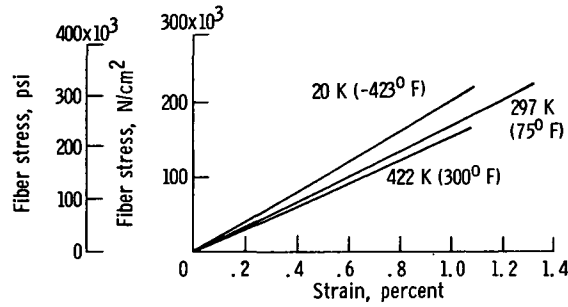


Figure 2. - Temperature effect on stress-strain diagram of PRD49-I/epoxy composite.

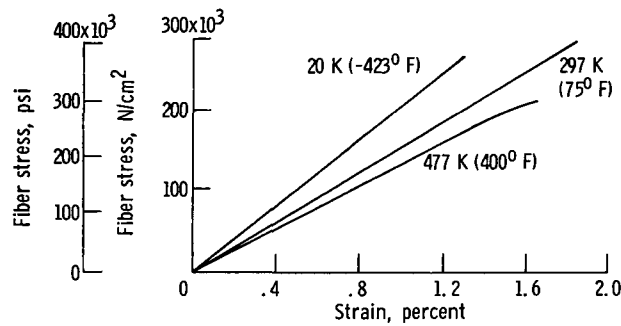


Figure 3. - Temperature effect on stress-strain diagram of PRD49-III/epoxy composite.

fibers, respectively. Corresponding tensile moduli were 16.7×10^6 and 15.0×10^6 N/cm² (24.2×10^6 and 21.7×10^6 psi). Short-time tensile strength at 477 K (400° F) for the type III fiber was about 73 percent of the 297 K (75° F) tensile strength. At 20 K (-423° F) the strength was about 91 percent of the 297 K (75° F) tensile strength. However, at 77 K (-320° F) the tensile strength was lower than at either 297 K (75° F) or 20 K (-423° F). The fiber tensile modulus for the type III fiber ranged from 20.7×10^6 N/cm² (30.0×10^6 psi) at 20 K (-423° F) to 12.4×10^6 N/cm² (19.5×10^6 psi) at 477 K (400° F). The fiber tensile strength and modulus of type III are shown as a function of temperature in figure 4.

An interesting observation is that the moduli of PRD49 fibers increase appreciably with decreasing test temperature. This is in contrast to inorganic fibers that do not exhibit any significant change in modulus with temperature. This modulus increase might be attributed to the fact that polymeric materials become extremely brittle and more rigid at cryogenic temperatures. In certain applications the higher cryogenic modulus would be advantageous. For example, in strain-limited filament-wound pressure vessels with metallic liners or in fiber-overwrapped metallic pressure vessels, at cryogenic temperatures a higher stress could be realized from the fiber for a given strain.

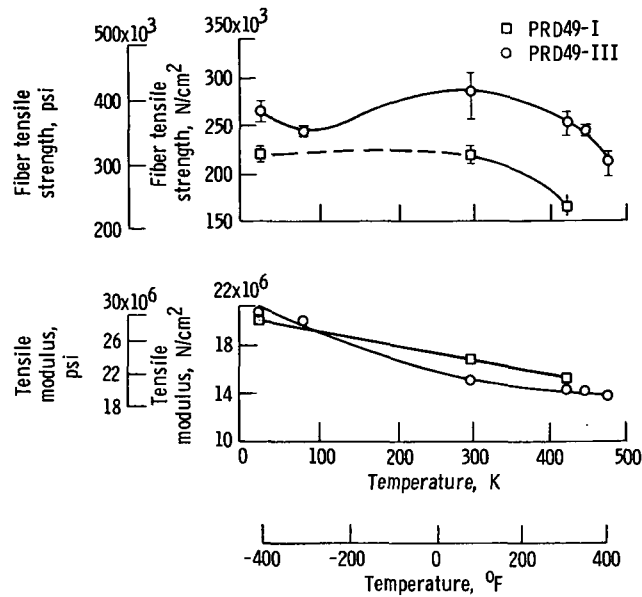


Figure 4. - Fiber tensile strength and modulus of PRD-49-I and PRD49-III/epoxy composites as function of temperature.

Creep Properties

The creep behavior of polymeric materials is an important factor to be considered when using polymers in structural applications. Generally, polymers exhibit creep properties that are markedly accelerated by elevated temperatures. Because PRD49 is a polymeric fiber, its long-term creep properties at ambient and elevated temperatures were investigated.

Initial creep studies were made on loop specimens of PRD49-I fiber described in the section MATERIALS AND FABRICATION. The results are shown in figure 5, where percent total strain is plotted as a function of time to 1000 hours. At 297 K (75° F) and

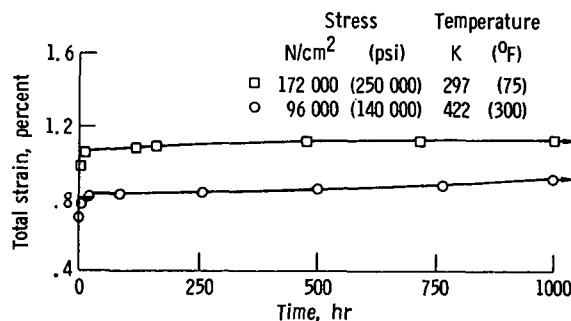


Figure 5. - Creep-time relations for PRD49-I/epoxy composite at various stress and temperature levels.

at a fiber stress of $172\,500\text{ N/cm}^2$ ($250\,000\text{ psi}$), which is approximately 80 percent of the 297 K (75° F) ultimate tensile strength, the specimen experiences an accelerated primary creep of about 0.1 percent within the first few hours after initial loading. This is followed by secondary creep at an essentially constant creep rate for the 1000-hour test duration. The secondary creep amounted to about 0.05 percent for the 1000-hour duration. At 422 K (300° F) and at a fiber stress of $96\,500\text{ N/cm}^2$ ($140\,000\text{ psi}$), the creep behavior is similar to that at 297 K (75° F), with the exception of a higher secondary creep rate at the lower stress level. The total creep elongation was about 0.2 percent. Comparing the two tests shows that temperature influences the creep rate of PRD49 fibers. Attempts to load the loop specimens at higher stress levels at 422 K (300° F) resulted in fracture of the specimen within the first few hours of the test. The failure location was usually at the ends of the specimen, where the fibers looped over the spool pieces. Creep tests using the loop specimen were discontinued. Creep studies on PRD49-III were performed with the unidirectional tensile specimen previously described.

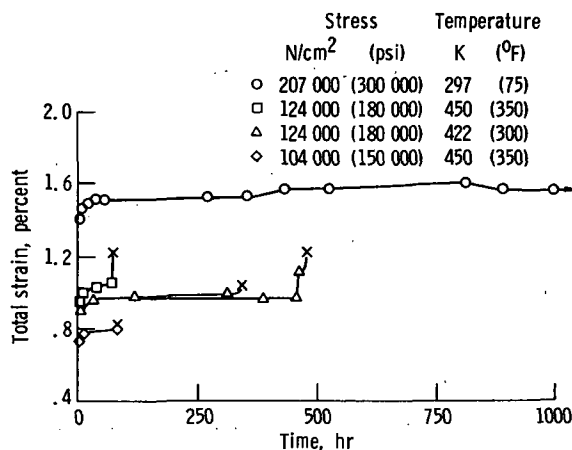


Figure 6. - Creep-time relations for PRD49-III/epoxy composite at various stress and temperature levels.

Figure 6 shows the percent of total strain as a function of time for PRD49-III. The fiber at 297 K (75° F) sustained a tensile stress of $207\,000\text{ N/cm}^2$ ($300\,000\text{ psi}$) (approximately 71 percent of the 297 K (75° F) ultimate tensile strength) for the 1000-hour test duration. The type III fiber appears to have creep behavior similar to that of type I fiber (fig. 5) at 297 K (75° F). However, some variations in creep were noted at 297 K (75° F) during the 1000-hour duration. Although no systematic records were made of the humidity throughout the test, it was noted that creep increased during periods of high humidity and experienced some recovery during periods of lower humidity. At

422 K (300° F) and at a stress level of 124 300 N/cm² (180 000 psi), one specimen failed in 460 hours and a second specimen failed in 375 hours. In both specimens the failures were preceded by progressive fiber failure extending over a period of several hours. The progressive failure was accompanied by an apparent increase in strain, as indicated in figure 6. Prior to the onset of fiber failure the creep behavior was similar to that of the 297 K (75° F) test. At 450 K (350° F) the maximum time to failure for the type III fiber was 54 hours at a stress level of 124 300 N/cm² (180 000 psi) and 93 hours at 103 500 N/cm² (150 000 psi). Failure of the fibers at elevated temperatures would imply stress rupture; however, it is possible that resin degradation also contributes to the failure process.

SUMMARY OF RESULTS

The following results of strength and creep properties were obtained from an investigation of PRD49-I and -III fibers in an epoxy matrix:

1. The fiber tensile strength at 297 K (75° F) was 221 000 and 294 000 N/cm² (320 000 and 425 000 psi) for type I and type III, respectively. Corresponding tensile moduli were 16.7×10⁶ and 15.0×10⁶ N/cm² (24.2×10⁶ and 21.7×10⁶ psi).
2. Short-time tensile-strength retention at 477 K (400° F) for the type III fiber was about 73 percent of the 297 K (75° F) tensile strength. At 20 K (-423° F) the strength retention was about 90 percent of the 297 K (75° F) tensile strength.
3. The fiber tensile modulus for the type III fiber ranged from 20.7×10⁶ N/cm² (30×10⁶ psi) at 20 K (-423° F) to 13.4×10⁶ N/cm² (19.5×10⁶ psi) at 477 K (400° F).
4. At 297 K (75° F) the type I and type III fibers sustained tensile stresses of 173 000 and 207 000 N/cm² (250 000 and 300 000 psi), respectively, for 1000 hours without failure. For both fiber types, the total creep elongation was about 0.15 percent during the 1000-hour test.
5. At 422 K (300° F), type I fiber sustained a 96 600 N/cm² (140 000 psi) tensile stress for 1000 hours without failure; type III failed in 460 hours at a tensile stress of 124 000 N/cm² (180 000 psi). The total creep elongation of type I in the 1000-hour test at 422 K (300° F) was about 0.20 percent.
6. At 450 K (350° F) the type III fiber at a tensile stress of 103 700 N/cm² (150 000 psi) failed in 93 hours; however, no excessive creep occurred until incipient progressive fiber failure concluded the test.
7. Depending on the test conditions, both fiber types showed typical accelerated primary creep behavior within the initial 25 hours. This was followed by secondary creep that occurred at a much lower rate.

8. At 297 K (75⁰ F), humidity appeared to have a minor affect on the creep behavior of PRD49 fiber.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 28, 1972,
501-21.

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